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PROBLEMS

9.1 Solutions of methanol and ethanol are substantially ideal.

(*a*) Compute the vapor-liquid equilibria for this system at 1 and at 5 atm abs pressure, and plot *xy* and *txy* diagrams at each pressure.

(*b*) For each pressure compute relative volatilities, and determine an average value.

(*c*) Using Eq. (9.2) with the average volatilities, compare the values of y^* at each value of x so obtained with those computed directly from the vapor pressures.

9.2 A 1000-kg batch of nitrobenzene is to be steam-distilled from a very small amount of a nonvolatile inpurity, insufficient to influence the vapor pressure of the nitrobenzene. The operation is to be carried out in a jacketed kettle fitted with a condenser and distillate receiver. Saturated steam at 35 kN/m^2 (5.1 lb_f/in^2) gauge is introduced into the kettle jacket for heating. The nitrobenzene is charged to the kettle at 25°C, and it is substantially insoluble in water.

Liquid water at 25°C is continuously introduced into the nitrobenzene in the kettle, to maintain a liquid water level. The mixture is distilled at atmospheric pressure. (*a*) At what temperature does

the distillation proceed? (*b*) How much water is vaporized? (*c*) How much steam must be condensed in the kettle jacket? Neglect the heat required to bring the still up to operating temperature. The heat capacity of nitrobenzene is 1382 J/kg · K, and its latent heat of vaporization can be determined by the methods of Chap. 7.

9.3 A solution has the following composition, expressed as mole fraction: ethane, 0.0025; propane, 0.25; isobutane, 0.185; *n*-butane, 0.560; isopentane, 0.0025. In the following, the pressure is 10 bars (145 lb_f/in^2) abs. Use equilibrium distribution coefficients from the Depriester nomograph (see Illustration 9.3).

(*a*) Calculate the bubble point.

(*b*) Calculate the dew point.

(*c*) The solution is flash-vaporized to vaporize 40 mol % of the feed. Calculate the composition of the products.

(*d*) The solution is differentially distilled to produce a residue containing 0.80 mole fraction *n*-butane. Calculate the complete composition of the residue and the percentage of the feed which is vaporized.

9.4 Vapor-liquid equilibrium data at 1 std atm abs, heats of solution, heat capacities, and latent heats of vaporization for the system acetone-water are as follows:

Mole fraction acetone in liquid, x	Integral heat of solution at 15°C, kJ/kmol soln	Equivalent mole fraction acetone in vapor, y^*	Vapor-liquid temperature, °C	Heat capacity at 17.2°C, kJ/(kg soln) · °C
0.001	0	0.00	100.0	4.187
0.01		0.253	91.7	4.179
0.02	−188.4	0.425	86.6	4.162
0.05	−447.3	0.624	75.7	4.124
0.10	−668.7	0.755	66.6	4.020
0.15	−770	0.798	63.4	3.894
0.20	−786	0.815	62.2	3.810
0.30	−719	0.830	61.0	3.559
0.40	−509	0.839	60.4	3.350
0.50	−350.1	0.849	60.0	3.140
0.60	−252.6	0.859	59.5	2.931
0.70		0.874	58.9	2.763
0.80		0.898	58.2	2.554
0.90		0.935	57.5	2.387
0.95		0.963	57.0	2.303
1.00		1.00	56.5	

t, °C	20	37.8	65.6	93.3	100
Heat capacity acetone, kJ/kg · °C	2.22	2.26	2.34	2.43	
Latent heat of vaporization, kJ/kg	1013	976	917	863	850

Compute the enthalpies of saturated liquids and vapors relative to pure acetone and water at 15°C and plot the enthalpy-concentration diagram, for 1 std atm abs. Retain for use in Probs. 9.5, 9.6, and 9.10.

9.5 A liquid mixture containing 60 mol % acetone, 40 mol % water, at 26.7°C (80°F), is to be continuously flash-vaporized at 1 std atm pressure, to vaporize 30 mol % of the feed.

(*a*) What will the composition of the products and the temperature in the separator be if equilibrium is established?

(*b*) How much heat, kJ/mol of feed, is required?

Ans.: 13 590.

(*c*) If the products are each cooled to 26.7°C, how much heat, kJ/mol of feed, must be removed from each?

9.6 A saturated vapor at 1 std atm pressure, containing 50 mol % acetone and 50 mol % water, is subject to equilibrium condensation to yield 50 mol % of the feed as liquid. Compute the equilibrium vapor and liquid compositions, the equilibrium temperature, and the heat to be removed, J/kmol of feed.

9.7 The liquid solution of Prob. 9.5 is differentially distilled at 1 atm pressure to vaporize 30 mol % of the feed. Compute the composition of the composited distillate and the residue. Compare with the results of Prob. 9.5.

Ans.: $y_D = 0.857$.

9.8 A jacketed kettle is originally charged with 30 mol of a mixture containing 40 mol % benzene, 60 mol % toluene. The vapors from the kettle pass directly to a total condenser, and the condensate is withdrawn. Liquid of the same composition as the charge is continuously added to the kettle at the rate of 15 mol/h. Heat to the kettle is regulated to generate vapor at 15 mol/h, so that the total molar content of the kettle remains constant. The mixture is ideal, and the average relative volatility is 2.51. The distillation is essentially differential.

(*a*) How long will the still have to be operated before vapor containing 50 mol % benzene is produced, and what is the composition of the composited distillate?

Ans.: 1.466 h, 0.555 mole fraction benzene.

(*b*) If the rate at which heat is supplied to the kettle is incorrectly regulated so that 18 mol/h of vapor is generated, how long will it take until vapor containing 50 mol % benzene is produced?

Ans.: 1.08 h.

9.9 An open kettle contains 50 kmol of a dilute aqueous solution of methanol, mole fraction methanol = 0.02, at the bubble point, into which steam is continuously sparged. The entering steam agitates the kettle contents so that they are always of uniform composition, and the vapor produced, always in equilibrium with the liquid, is led away. Operation is adiabatic. For the concentrations encountered it may be assumed that the enthalpy of the steam and evolved vapor are the same, the enthalpy of the liquid in the kettle is essentially constant, and the relative volatility is constant at 7.6. Compute the quantity of steam to be introduced in order to reduce the concentration of methanol in the kettle contents to 0.001 mole fraction.

Ans.: 20.5 kmol.

Continuous fractionation: Savarit-Ponchon method

9.10 An acetone-water solution containing 25 wt % acetone is to be fractionated at a rate of 10 000 kg/h at 1 std atm pressure, and it is planned to recover 99.5% of the acetone in the distillate at a concentration of 99 wt %. The feed will be available at 26.7°C (80°F) and will be preheated by heat exchange with the residue product from the fractionator, which will in turn be cooled to 51.7°C (125°F). The distilled vapors will be condensed and cooled to 37.8°C (125°F) by cooling water entering at 26.7°C and leaving at 40.6°C. Reflux will be returned at 37.8°C, at a reflux ratio $L_0/D = 1.8$. Open steam, available at 70 kN/m^2, will be used at the base of the tower. The tower will be insulated to reduce heat losses to negligible values. Physical property data are given in Prob. 9.4. Determine:

(*a*) The hourly rate and composition of distillate and reflux.

(*b*) The hourly condenser heat load and cooling water rate.

(*c*) The hourly rate of steam and residue and the composition of the residue.

(*d*) The enthalpy of the feed as it enters the tower and its condition (express quantitatively).

(*e*) The number of theoretical trays required if the feed is introduced at the optimum location.

Use large-size graph paper and a sharp pencil.

Ans.: 13.1.

(*f*) The rates of flow, kg/h, of liquid and vapor at the top, at $x = 0.6$, 0.1, 0.025, and at the bottom tray. For a tower of uniform diameter, the conditions on which tray control the diameter, if the criterion is a 75% approach to flooding?

9.11 In a certain binary distillation, the overhead vapor condenser must be run at a temperature requiring mild refrigeration, and the available refrigeration capacity is very limited. The reboiler temperature is very high, and the available high-temperature heat supply is also limited. Consequently the scheme shown in Fig. 9.56 is adopted, where the intermediate condenser E and reboiler S operate at moderate temperatures. Condensers C and E are total condensers, delivering liquids at their bubble points. Reboiler S is a total vaporizer, delivering saturated vapor. Heat losses are negligible, $D = W$, the external reflux ratio $R = L_0/D = 1.0$, and other conditions are shown on the figure. The system is one which follows the McCabe-Thiele assumptions ($H_L x$ and $H_G y$ curves are straight and parallel). Feed is liquid at the bubble point.

(*a*) Per mole of feed, compute the rates of flow of all streams, the relative size of the two condenser heat loads, and the relative size of the two vaporizer heat loads.

(*b*) Establish the coordinates of all difference points. Sketch the Hxy diagram and locate the difference points, showing how they are related to the feed. Show the construction for trays.

(*c*) Sketch the xy diagram, show all operating lines, and locate their intersections with the 45° diagonal. Show the construction for trays.

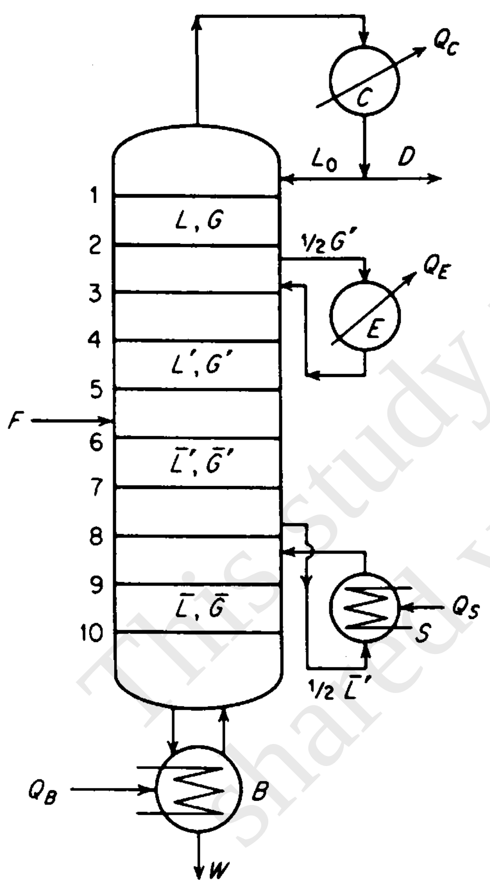


Figure 9.56 Arrangement for Prob. 9.11.

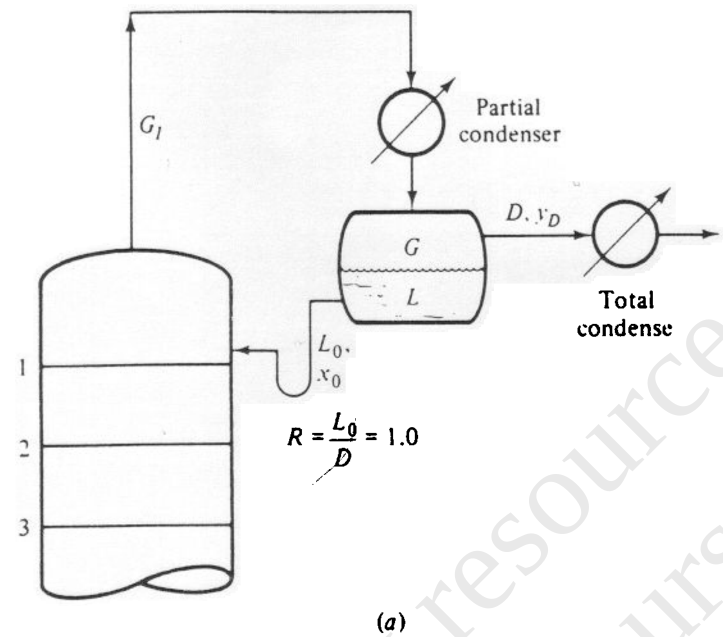
9.12 A distillation column is operated at a temperature below that of the surroundings (as in the distillation of air). Imperfect insulation causes the entire enriching section to be subject to a heat leak inward of Q_E J/s, while the entire stripping section is t to a heat leak inward of Q_S J/s. Aside from the heat leaks, the system has all the characteristics which satisfy the McCabe-Thiele requirements.

(*a*) Sketch the Hxy and the xy diagrams, the of all the operating curves on the xy diagram concave upward or downward?

(*b*) For a given condenser heat load, explain which is better: to design a new column with the inward heat leak minimized, or to take any possible advantage of a heat leak. Consider the reboiler heat load free, available from the surroundings.

(*c*) Suppose the column with the inward heat leak is operated at full reboiler heat load, limited by the available heat-transfer surface. The column is then insulated thoroughly, essentially eliminating the heat leak, but without provision to alter the heat load of the reboiler or otherwise to alter the operation. Explain whether the separation obtained will be better than, worse than, or the same as, before the insulation was installed.

9.13 A distillation tower for a binary solution was designed with a partial condenser, using $L_0/D = 1.0$, as in Fig. 9.57*a*. When built, the piping was installed incorrectly, as in Fig. 9.57*b*. It was then argued that the error was actually a fortunate one, since the richer reflux would provide a withdrawn distillate richer than planned. The mixture to be distilled has all the characteristics of one obeying McCabe-Thiele assumptions. For a reflux ratio = 1.0, reflux at the bubble point, and the



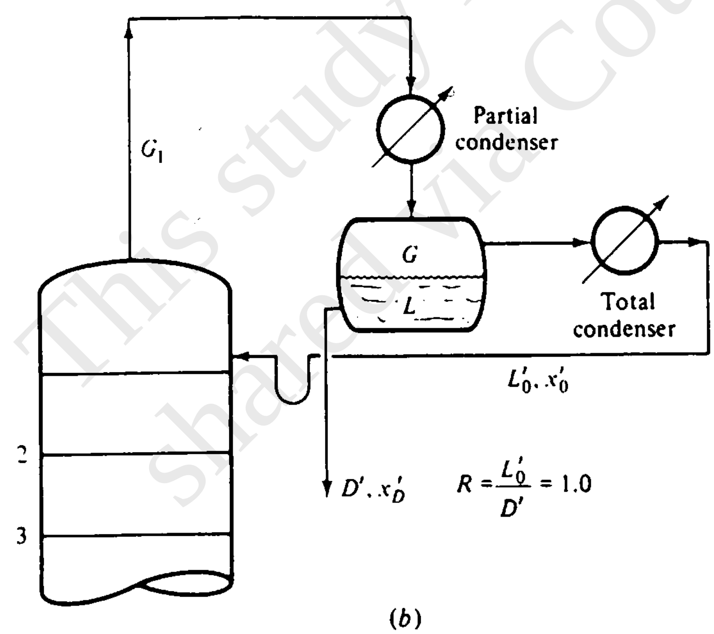


Figure 9.57 Arrangements for Prob. 9.13.

arrangement of Fig. 9.57*b*:

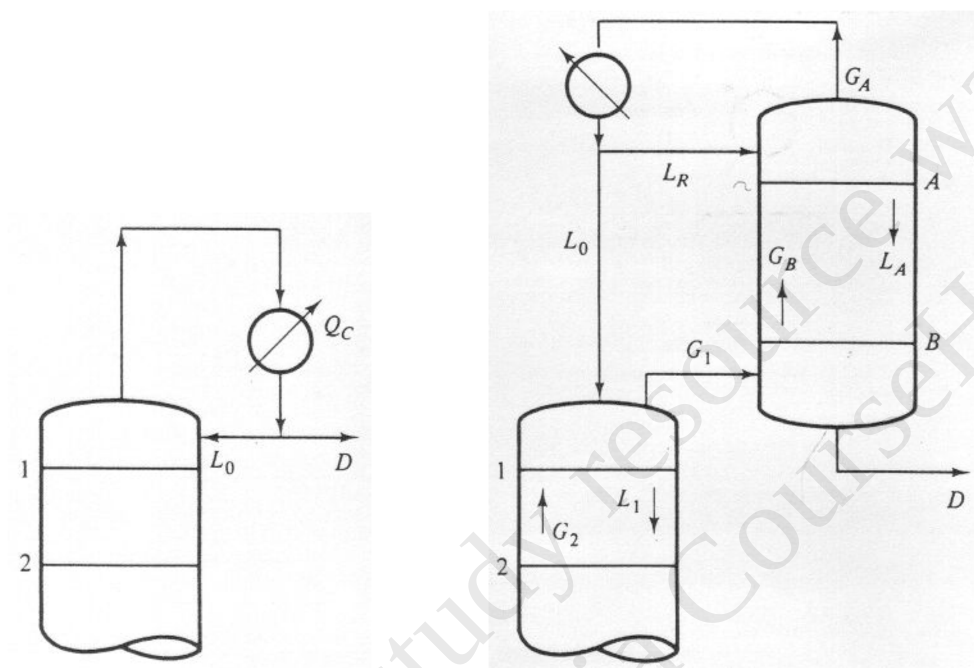
(*a*) Sketch the Ponchon-Savarit and *xy* diagrams for the parts of the columns indicated in the figure. Label the indicated vapor and liquid concentrations.

(*b*) Establish whether or not the distillate will be richer than originally planned.

9.14 Figure 9.58*a* shows a few of the top trays of a conventionally arranged fractionator. It has been suggested that a richer distillate might be obtained by the scheme shown in Fig. 9.58*b*, because of the richer reflux to tray 1. All trays are theoretical. The condenser is a total condenser, delivering liquid at the bubble point in both cases. For both arrangements, $L_0/D = L_0/L_R = 1.0$.

(*a*) For a binary mixture which in every respect follows the McCabe-Thiele simplifying assumptions, sketch fully labeled *Hxy* and *xy* diagrams for the scheme of Fig. 9.58*b*. Mark on the *Hxy* diagram the location of every stream on Fig. 9.58*b* and the required difference points. Show construction lines and tie lines.

(*b*) Prove that for all otherwise similar circumstances the scheme of Fig. 9.58*b* in fact delivers a *leaner* distillate than that of Fig. 9.58*a*.



(*a*) (*b*)

Figure 9.58 Arrangements for Prob. 9.14.

Continuous fractionation: McCabe-Thiele method

9.15 A solution of carbon tetrachloride and carbon disulfide containing 50 wt% each is to be continuously fractionated at standard atmospheric pressure at the rate 4000 kg/h. The distillate product is to contain 95 wt % carbon disulfide, the residue 0.5%. The feed will be 30 mol % vaporized before it enters the tower. A total condenser will be used, and reflux will be returned at the bubble point. Equilibrium data ("The Chemical Engineers' Handbook," 4th ed., p. 13-5), x, y = mole fraction CS_2:

t, °C	x	y^*	t, °C	x	y^*
76.7	0	0	59.3	0.3908	0.6340
74.9	0.0296	0.0823	55.3	0.5318	0.7470
73.1	0.0615	0.1555	52.3	0.6630	0.8290
70.3	0.1106	0.2660	50.4	0.7574	0.8780
68.6	0.1435	0.3325	48.5	0.8604	0.9320
63.8	0.2585	0.4950	46.3	1.000	1.000

(*a*) Determine the product rates, kg/h.

(*b*) Determine the minimum reflux ratio.

(c) Determine the minimum number of theoretical trays, graphically and by means of Eq. (9.85).

(*d*) Determine the number of theoretical trays required at a reflux ratio equal to twice the minimum and the position of the feed tray.

Ans.: 12.5 theoretical trays.

(*e*) Estimate the overall tray efficiency of a sieve-tray tower of conventional design and the number of real trays.

(*f*) Using the distillate temperature as the base temperature, determine the enthalpy of the feed, the products, and the vapor entering the condenser. Determine the heat loads of the condenser and reboiler. Latent and specific heats are available in "The Chemical Engineers' Handbook," 5th ed., pp. 3-116 and 3-129.

9.16 A solution of carbon tetrachloride and carbon disulfide containing 50 wt % of each is to be continuously fractionated to give a distillate and residue analyzing 99.5 and 0.5 wt % carbon disulfide, respectively. Feed will be available at the bubble point, and reflux will be returned at the bubble point.

There is available a sieve-tray tower of conventional design, suitable for use at 1 std atm pressure abs. The diameter is 0.75 m (30 in), and it contains 26 identical cross-flow trays at a spacing of 0.50 m (20 in). A feed nozzle is available only for the tenth tray from the top. Each tray contains a straight weir, 0.46 m (18 in) long, extending 65 mm (2.5 in) from the tray floor. The perforated area is 12% of the active area of the perforated sheet. Adequate condenser and reboiler will be supplied. Equilibrium data are listed in Prob. 9.15.

The listed products represent the minimum purities acceptable, and higher purities at the expense of additional heat load or reduced capacity are not warranted. Estimate the largest feed rate which the column can reasonably be expected to handle.

9.17 Refer to Fig. 9.46, the McCabe-Thiele diagram for separating partially miscible mixtures. Determine the coordinates of the point of intersection of the enriching-section operating line of tower I and the 45° diagonal of the diagram.

9.18 An aqueous solution of furfural contains 4 mol % furfural. It is to be continuously fractionated at 1 std atm pressure to provide solutions containing 0.1 and 99.5 mol % furfural, respectively. Feed is liquid at the bubble point. Equilibrium data ("The Chemical Engineers' Handbook," 4th ed., p. 13-5), x, y = mole fraction furfural:

t, °C	x	y^*	t, °C	x	y^*
100.	0	0	100.6	0.80	0.11
98.56	0.01	0.055	109.5	0.90	0.19
98.07	0.02	0.080	122.5	0.92	0.32
97.90	0.04	0.092	146.0	0.94	0.64
97.90	0.092	0.092	154.8	0.96	0.81
97.90	0.50	0.092	158.8	0.98	0.90
98.7	0.70	0.095	161.7	1.00	1.00

(*a*) Arrange a scheme for the separation using kettle-type reboilers, and determine the number of theoretical trays required for a vapor boilup rate of 1.25 times the minimum for an infinite number of trays.

(*b*) Repeat part (*a*) but use open steam at essentially atmospheric pressure for the tower delivering the water-rich product, with 1.25 times the minimum steam and vapor boilup rates.

(*c*) Which scheme incurs the greater furfural loss to the water-rich product?

9.19 An aniline-water solution containing 7 wt % aniline, at the bubble point, is to be steam-stripped at a rate of 1000 kg/h in a tower packed with 38-mm (1.5-in) Berl saddles with open steam to remove 99% of the aniline. The condensed overhead vapor will be decanted at 98.5°C, and the water-rich layer returned to the column. The aniline-rich layer will be withdrawn as distillate product. The steam rate will be 1.3 times the minimum. Design the tower for a pressure drop of 400

(N/m^2)/m at the top. *Note:* For concentrations where the equilibrium curve is nearly straight, it should be possible to use overall numbers and heights of transfer units.

Ans.: 6.5 m of packing.

Data: at 98.5°C, the solubility of aniline in water is 7.02 and 89.90 wt % aniline. The vapor-liquid equilibria at 745 mmHg, at which pressure the tower will be operated, are [Griswold et al., *Ind. Eng. Chem.*, 32, 878 (1940)]:

x = mole fraction aniline	0.002	0.004	0.006	0.008	0.010	0.012	Two liquid phases, 98.5°C
y^* = mole fraction aniline	0.01025	0.0185	0.0263	0.0338	0.03575	0.03585	0.0360

9.20 A distillation column is to be fed with the following solution:

$$n\text{-}C_3H_8 = 0.06 \text{ mole fraction}$$
$$i\text{-}C_4H_{10} = 0.65$$
$$n\text{-}C_4H_{10} = 0.26$$
$$n\text{-}C_5H_{12} = \frac{0.03}{1.00}$$

The distillate is to contain no more than 0.05 mole fraction n-C_4H_{10} and essentially no C_5H_{12}. The bottoms will contain no more than 0.05 mole fraction i-C_4H_{10} and essentially no C_3H_8. The overhead vapor will be completely condensed at its bubble point, 37.8°C (100°F), with reflux to the top tray at the rate 2 kmol reflux/kmol distillate withdrawn. Compute the column pressure to be used.

9.21 The following feed at the bubble point at 827 kN/m^2 (120 lb$_f$/in^2) abs, is to be fractionated at that pressure to provide no more than 7.45% of the n-C_5H_{12} in the distillate and no more than 7.6% of the n-C_4H_{10} in the bottoms, with a total condenser and reflux returned at the bubble point:

Component	x_F	60°C	90°C	120°C
n-C_3H_8	0.05			
m		2.39	3.59	5.13
H_G		33 730	36 290	39 080
H_L		20 350	25 800	31 050
i-C_4H_{10}	0.15			
m		1.21	1.84	2.87
H_G		40 470	43 960	47 690
H_L		24 050	28 800	34 560
n-C_4H_{10}	0.25			
m		0.80	1.43	2.43
H_G		43 730	46 750	50 360
H_L		24 960	30 010	35 220
i-C_5H_{12}	0.20			
m		0.39	0.72	1.29
H_G		51 870	55 270	59 430
H_L		29 080	34 610	40 470
n-C_5H_{12}	0.35			
m		0.20	0.615	1.13
H_G		54 310	57 680	61 130
H_L		29 560	35 590	41 710

The table provides Henry's-law constants and enthalpies expressed as kJ/kmol, from the same sources and using the same base as the data of Illustration 9.13.

Compute the minimum reflux ratio, the minimum number of theoretical trays, and for a reflux ratio $R = 2.58$ estimate the product analyses, condenser and reboiler heat loads, vapor and liquid rates per mole of feed throughout, and the number of theoretical trays.

Ans.: 8 theoretical trays + reboiler.

9.22 The following feed at 60°C (140°F), 2070 kN/m^2 (300 lb$_f$/in^2) abs, is to be fractionated at that pressure so that the vapor distillate product contains 0.913 percent of the isopentane and the bottoms 0.284 percent of the isobutane:

$$
\begin{aligned}
CH_4 &= 0.50 \text{ mole fraction} \\
C_3H_8 &= 0.03 \\
i\text{-}C_4H_{10} &= 0.10 \\
n\text{-}C_4H_{10} &= 0.15 \\
i\text{-}C_5H_{12} &= 0.07 \\
n\text{-}C_5H_{12} &= 0.05 \\
n\text{-}C_6H_{14} &= 0.10 \\
& \overline{1.00}
\end{aligned}
$$

Compute: (*a*) The minimum reflux ratio and (*b*) the minimum number of trays. At a reflux ratio = 2.0, estimate (*c*) the product analyses and (*d*) the number of theoretical trays by the correlations of Gilliland [17], Erbar and Maddox [12], Brown and Martin [5], and Strangio and Treybal [59].